

NASA Technical Paper 1035

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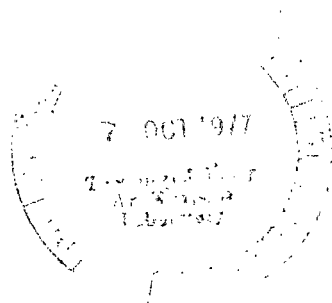


Friction and Wear Behavior of Single-Crystal Silicon Carbide in Contact With Titanium

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National Aeronautics
and Space Administration

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1977

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by Kazuhisa Miyoshi* and Donald H. Buckley

Lewis Research Center

SUMMARY

An investigation was conducted to examine the friction and wear behavior of single-crystal silicon carbide in contact with titanium and the nature of interfacial transfer. Sliding friction experiments were conducted with a polycrystalline titanium rider sliding on a single-crystal silicon carbide (0001) surface in the $\langle 10\bar{1}0 \rangle$ directions. Sliding was both in argon at atmospheric pressure and in high vacuum (10^{-8} N/m²). Applied loads varied up to 50 grams. Auger emission spectroscopy analysis was used to monitor the surface in high vacuum.

The results of the investigation indicate that the friction coefficient is greater in vacuum than in argon and that this is due to the greater adhesion or adhesive transfer in vacuum. Thin films of silicon carbide transferred to the titanium and titanium also adhered to the silicon carbide both in argon at atmospheric pressure and in high vacuum. Cohesive bonds fractured on both the silicon carbide and titanium surfaces. Wear debris of silicon carbide created by fracture plowed the silicon carbide surface in a plastic manner. The friction characteristics of titanium in contact with silicon carbide were sensitive to the surface asperities on silicon carbide, and the friction coefficients were higher for a rough surface of silicon carbide than for a smooth one. The difference in friction results was due to plastic deformation (plowing of titanium).

INTRODUCTION

For many years, it has been known that silicon carbide has outstanding mechanical and physical properties. Silicon carbide is at the same time a very old ceramic (grinding wheels) and also one of the newest (turbine blades, vanes, and shrouds in gas turbine engines). The considerable recent interest in silicon carbide in the field of tribology has been stimulated mainly by one of its key properties, that is, its high wear resistance

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under a variety of exacting environmental conditions. But, the tribophysical properties of silicon carbide in contact with metals, ceramics, and polymers are not clearly understood. Very little experimental work has been done with silicon carbide.

Silicon carbide in contact with silicon carbide exhibits lower friction than unlubricated metals and retains it to much higher temperatures (refs. 1 and 2). Furthermore, the effect of crystallographic orientation and load on the Knoop microhardness of single-crystal silicon carbide has been investigated (ref. 3). Nevertheless, there is a great lack of fundamental information about the mechanism of friction and the mechanical behavior.

The present investigation was conducted to examine the friction and wear behavior of single-crystal silicon carbide in contact with titanium and the nature of interfacial transfer. All experiments were conducted with light loads of 5 to 50 grams, at a sliding velocity of 0.7 mm/min in a vacuum of 10^{-8} N/m² or at a sliding velocity of 3 mm/min in argon, on the (0001) basal plane in the $\langle 10\bar{1}0 \rangle$ directions. All experiments were conducted at room temperature.

MATERIALS

The single-crystal silicon carbide platelets in these experiments were a 99.9-percent-pure compound of silicon and carbon. Silicon carbide had a hexagonal crystal structure with the most commonly occurring unit cell dimensions of $a = 3.0817 \text{ \AA}$ and $c = 15.1183 \text{ \AA}$. The unit cell contained two interpenetrating close-packed atomic arrays, one of silicon and the other of carbon displaced by $1/4$ -layer spacing along the c -axis. The silicon atoms thus occupied the tetrahedral locations in the array of carbon atoms and conversely. Hence, the basic unit of the structure can be considered to be a plane of tetrahedra, arbitrarily SiC_4 or CSi_4 . The c -direction was perpendicular to the sliding interface, with the basal planes therefore parallel to the interface.

The titanium polycrystal was 99.97 percent pure and had a hexagonal close-packed crystal structure.

APPARATUS

Two apparatuses were used in this investigation. One was a system capable of applying load and measuring friction in argon. The mechanism for measuring friction is shown schematically in figure 1(a). The beam contained one flat machined normal to the direction of friction application. The end of the rod contained the polycrystalline titanium pin specimen. Load was applied by placing deadweights on a pan on top of the

rod. Under an applied load, the friction force was sensed by the strain gage.

The second apparatus was a vacuum system capable of measuring adhesion, load, and friction and capable of Auger and low-energy electron diffraction (LEED) surface analysis. The mechanism for applying load and measuring adhesion and friction is shown schematically in figure 1(b). A gimbal-mounted beam projected into the vacuum system. The beam contained two flats machined normal to each other with strain gages mounted thereon. The end of the rod contained the polycrystalline titanium pin specimen. The load applied by moving the beam toward the flat plate was measured by the strain gage. Tangential motion of the pin along the flat plate was accomplished through the gimbal assembly. Under an applied load, the friction force was sensed by the strain gage normal to that used to measure the applied load. Pin sliding was in the vertical direction of figure 1(b).

The vacuum apparatus in which the components of figure 1(b) were contained also had a LEED diffraction system and an Auger spectrometer. The electron beam of both could be focused on any flat-plate site desired. This was accomplished with a flat-plate manipulation device. The vacuum system was a conventional vacsorb and ion-pumped system capable of readily achieving pressures of $1.33 \times 10^{-8} \text{ N/m}^2$ (10^{-10} torr) as measured by a nude ionization gage within the specimen chamber. Sublimation pumping was also used to more rapidly achieve the pressure desired.

EXPERIMENTAL PROCEDURE

The polycrystalline titanium surface was hemispherical and was polished with approximately 1- μm -diameter aluminum oxide (Al_2O_3) powder. The radius of the titanium rider was 0.79 mm (1/32 in.). The titanium was then chemically polished. The surfaces were rinsed with water and 200-proof ethyl alcohol.

The single-crystal silicon carbide surfaces were used both in the original as-grown platelet condition (rough surfaces) and as surfaces mechanically polished with a 1- μm Al_2O_3 powder (smooth surfaces) (fig. 2).

For the experiments in vacuum, the specimens were placed in the vacuum chamber and the system was evacuated. It was baked out overnight, after which the pressure was in the 10^{-8} - N/m^2 range. Argon gas was bled into the vacuum system to a pressure of 1.3 N/m^2 . A 1000-volt direct-current potential was applied to the specimen, and it was sputter bombarded for 30 minutes. After sputtering, the surface was examined with Auger emission spectroscopy to establish the surface cleanliness. If the surface was not clean, the sputtering process was repeated. The silicon carbide was placed in a tantalum harness for surface sputter cleaning. All friction experiments in vacuum were conducted with the system reevacuated to a pressure of 10^{-8} N/m^2 .

RESULTS AND DISCUSSION

Friction and Wear in Argon at Atmospheric Pressure

Friction of titanium sliding on silicon carbide. - Sliding friction experiments were conducted with the titanium specimen in contact with both smooth and rough silicon carbide (0001) surfaces. Surface profiles are shown in figure 2. The friction coefficients measured at various loads for the titanium rider in sliding contact with the silicon carbide are presented in figure 3. The data of figure 3 show marked differences in friction behavior with the smooth and rough surfaces. The rough surface of silicon carbide produces higher measured friction coefficients. Surface asperities, as shown in figure 2, also have a marked effect on the friction force traces (fig. 4). The friction force traces for the smooth surface of silicon carbide are characterized by randomly fluctuating behavior, with no evidence of stick-slip behavior. On the other hand, the traces for the rough surface are characterized by marked stick-slip behavior.

Wear of titanium sliding on smooth silicon carbide. - Titanium sliding on smooth silicon carbide results in the transfer of silicon carbide to the titanium, as shown in figure 5. Figures 5(a) and (b) are scanning electron micrographs of the rider wear scar on titanium after it slid on a smooth carbide surface. In figure 5(a) to the left-top of the wear scar is the beginning of the contact area with silicon carbide, the midsection in the figure is the end of the contact area. A scanning electron micrograph of the end of the wear scar, where many transfer films of silicon carbide are evident, and an X-ray energy-dispersive analysis for silicon on the titanium surface are shown in figures 5(b) and (c), respectively. The concentrations of white spots in figure 5(c) correspond to those locations in figure 5(b) where transfer is evident. The copious amount of thin silicon carbide film shown on the titanium in figure 5 was the result of a single pass of the titanium rider across the silicon carbide flat surface. After these films were formed in sliding, that is, at the end of the wear scar, a large number of deep grooves appeared on the surface of the titanium, as shown in figure 6(a). These grooves were produced in a plastic manner by the plowing of hard asperities of silicon carbide transferred to the titanium. Such a transfer film is still evident at the beginning of the wear scar in the scanning electron micrograph (fig. 6(b)).

Examining the wear track on the silicon carbide surface caused by titanium sliding against it revealed evidence of fracture in silicon carbide at a higher load of 50 g (fig. 7) and transfer of titanium to the silicon carbide.

Wear of titanium sliding on rough silicon carbide. - When titanium slid on rough silicon carbide, a large number of plastically deformed grooves appeared on the titanium surface, and titanium was transferred to the randomly distributed asperities of the silicon carbide, as shown in figures 8 to 10. Figure 8 shows scanning electron micrographs of the rider wear scar created by titanium sliding on a rough silicon carbide surface

under a 30-g load (fig. 2(b)). Figure 9 contains a scanning electron micrograph and an X-ray energy-dispersive analysis of a wear scar caused by titanium sliding on a rough silicon carbide surface also under a 30-g load. No white spots in figure 9(b) correspond to those locations in figure 9(a) where transfer of silicon carbide to titanium is evident.

Figure 10 reveals that most contact asperities were covered with the adhered titanium. The number of stick-slip motions in figure 4(b) and the number of contact asperities were almost the same. These facts suggest that the increase in stick and friction shown in figure 4(b) represents the force required to overcome the cohesive bonds in the titanium adhered to individual asperities of silicon carbide. The slip reflects fracture of the cohesive bonds in the titanium. Grooves are formed in the wear scar by the plowing action of the hard asperities of silicon carbide (figs. 8 to 10). This type of sliding also is evidence of films of silicon carbide transferred to the titanium (fig. 9).

Friction and Wear in High Vacuum

Friction of titanium sliding on silicon carbide. - Auger emission spectroscopy analysis of the smooth and rough silicon carbide surfaces before sputter cleaning revealed the presence of surface contaminants. Silicon peaks were not clearly detected (figs. 11(a) and (c)). After these same silicon carbide surfaces were sputter cleaned by argon bombardment for 60 minutes, the Auger spectra of figures 11(b) and (d) were obtained. In these spectra, silicon and carbon peaks are clearly seen.

Sliding friction experiments were conducted with titanium in contact with both smooth and rough silicon carbide (0001) surfaces. The surface profiles are shown in figure 12. The number of asperities on a rough surface, in comparison with figure 2(b), was reduced by sputter cleaning. However, sharp asperities still remained on the rough surface.

The friction coefficients measured at various loads are presented in figure 13. There were also marked differences in friction behavior with surface asperities. The rough surface of silicon carbide produced the higher measured friction coefficient. The friction behavior with load was very similar to the friction properties in argon at atmospheric pressure; but a shift upward occurred in friction values.

However, the differences in friction force traces between smooth and rough surfaces were small, as shown in figure 14; and both traces are characterized by marked stick-slip behavior. This type of friction is anticipated where strong adhesion occurs at the interface. The stick-slip friction behavior in the traces represents the force required to tangentially overcome the cohesive bond of the titanium (fig. 15). The slip reflects fracture of the cohesive bond. Figure 15(a) clarifies the relation between the transfer of titanium to silicon carbide and the stick-slip behavior.

Wear of titanium sliding on smooth silicon carbide. - Sliding of titanium on smooth

silicon carbide results in both the transfer of silicon carbide to titanium (figs. 16 and 17) and the transfer of titanium to silicon carbide (fig. 18). Figures 16 and 17 show scanning electron micrographs of the rider wear scar on titanium after it slid on a smooth silicon carbide surface and X-ray dispersive analyses of the wear scar. The left side of figure 16 is the beginning of the wear scar and the right side is the end of the wear scar. Figure 16(a) reveals that silicon carbide wear debris was produced by friction, plowing the titanium and transferring to the titanium primarily at the end of the wear scar. Thus, fracture may be anticipated to occur readily in silicon carbide at the adhesive bond. Increasing the magnification (fig. 17) clarifies fractures formed in the silicon carbide wear debris and the features of the wear debris.

On the other hand, detailed examinations of the wear scar on the silicon carbide and the wear debris after sliding with titanium revealed evidence of both thin films of titanium transferred to the silicon carbide (fig. 18) and wear debris of silicon carbide covered with titanium (fig. 19). Wear debris of silicon carbide plows the silicon carbide surface itself, and the groove is produced in a plastic manner on the surface of silicon carbide, as shown by the arrows in figure 19(a).

In the silicon carbide wear track shown in figure 20, the silicon carbide has been fractured by the sliding. Figure 21 shows that the wear debris originates at the fractures and perpendicular to the sliding direction.

Wear of titanium sliding on rough silicon carbide. - With a rough surface, silicon carbide was transferred to the titanium and the silicon carbide fractured and plastically deformed. Figure 22 reveals the film of silicon carbide transferred to the titanium and a large number of deep grooves. These grooves are formed by the plowing of hard asperities of silicon carbide (fig. 12(b)). Figure 23 contains scanning electron micrographs of the silicon carbide wear track. Fracture and plastic deformation have been generated with sliding. These facts suggest that the grooves are produced by plowing of hard silicon carbide asperities transferred to the titanium. Furthermore, a transfer of titanium to silicon carbide was also confirmed.

Comparison of Results in Argon and Vacuum Environments

Figure 24 summarizes friction coefficients measured at various loads in both argon and high vacuum. The friction differences are significant. With a smooth surface of silicon carbide in argon both at atmospheric pressure and in high vacuum, adhesion plays an important role. With a rough surface of silicon carbide in both environments, friction is due to shearing adhesive bonds at the interface and cohesive bonds in titanium or silicon carbide and to plowing of the titanium by asperities of silicon carbide.

With a rough surface of silicon carbide in high vacuum, frictional energy may be also dissipated by the plastic deformation and fracture of silicon carbide (fig. 23).

The friction and wear data obtained with silicon carbide contacting titanium indicate that two wear mechanisms are involved when these materials are in sliding contact. Both adhesive and abrasive wear occur. In argon, with the lower surface contaminants, friction was greater for the rough surface than it was for the smooth surface. A difference in friction between the smooth and rough surfaces persisted in vacuum, but the friction coefficients for both surfaces were considerably higher in vacuum than in argon. The increase in friction observed with both surfaces in vacuum, when the surfaces were clean, reflects the increase in interfacial adhesive bonding when surface contaminants are removed. Thus, adhesion contributes to friction and wear.

The fact that a difference in friction coefficient for the smooth and rough surfaces exists in both environments, with the smooth surface exhibiting the lower value, indicates that a force in addition to adhesion is contributing to friction. That force is the force necessary for the asperities of the rough silicon carbide to plow through the titanium. It is the force that contributes to the generation of abrasive wear particles. Thus, the friction coefficients for the smooth surface in both environments reflect adhesion forces, while the difference in friction coefficient for the smooth and rough surfaces represents the effect of plowing or plastic deformation.

CONCLUSIONS

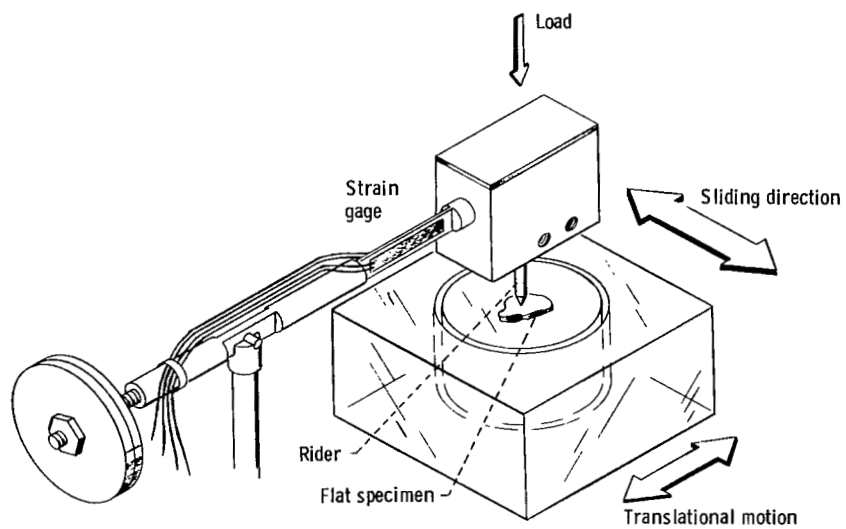
As a result of sliding friction experiments conducted with single-crystal silicon carbide in sliding contact with titanium, the following conclusions were drawn:

1. The friction coefficient in vacuum was greater than that in argon, and this was due to the greater adhesion or adhesive transfer in vacuum.
2. Both in argon at atmospheric pressure and in high vacuum, thin films of silicon carbide transferred to the titanium. Titanium also transferred to silicon carbide. The cohesive bonds on both the silicon carbide and titanium surfaces fractured as a result of sliding.
3. Silicon carbide wear debris caused by fracture plowed the silicon carbide surface in a plastic manner.
4. The friction characteristics of titanium in contact with silicon carbide were sensitive to the surface roughness of silicon carbide. The friction coefficients were higher with a rough surface of silicon carbide than with a smooth one. The difference in friction results was due to plastic deformation (plowing of titanium).

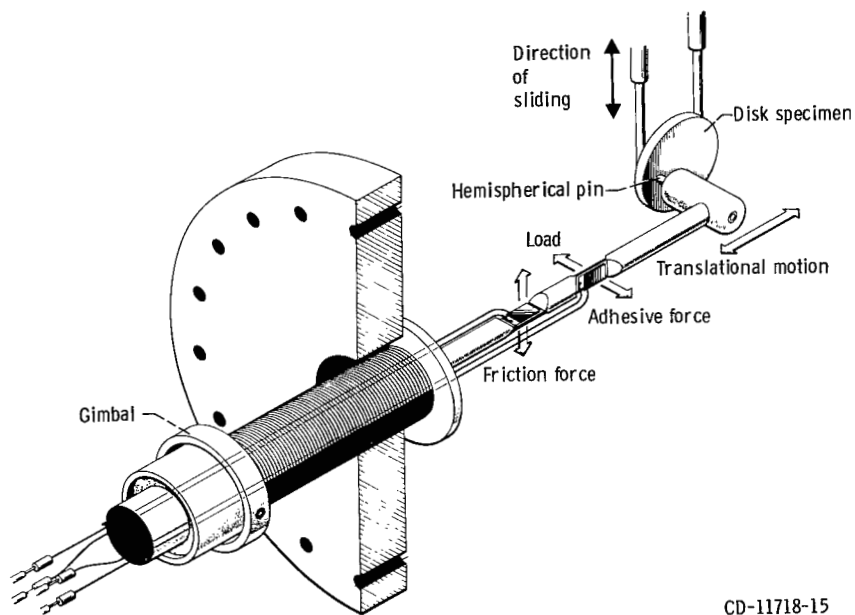
Lewis Research Center,
National Aeronautics and Space Administration,
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506-16.

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2. Brookes, C. A.; and Atkins, A. G.: The Friction and Hardness of Refractory Compounds. Fifth Plansee Seminar De Re Metallica on Metals for the Space Age. F. Benesovsky, ed., Metallwerk Plansee AG, 1965, pp. 712-720.
3. Adewoye, O. O.; et al.: Structural Studies of Surface Deformation in MgO, SiC and Si_3N_4 . Cambridge Univ., England (AD-A008993), 1974.



(a) Apparatus used in argon.



(b) High-vacuum apparatus.

Figure 1. - Friction and wear apparatus.

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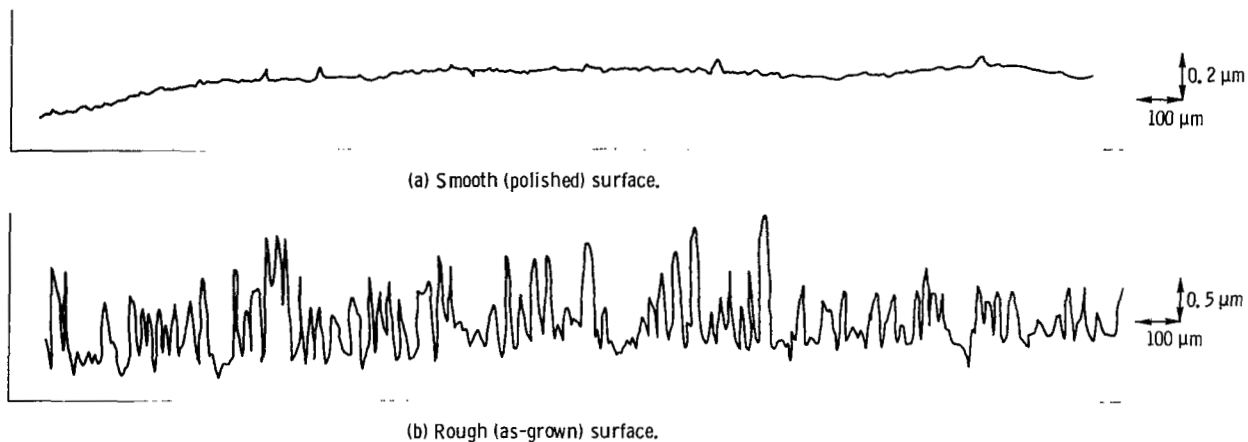


Figure 2. - Surface profile curves for silicon carbide.

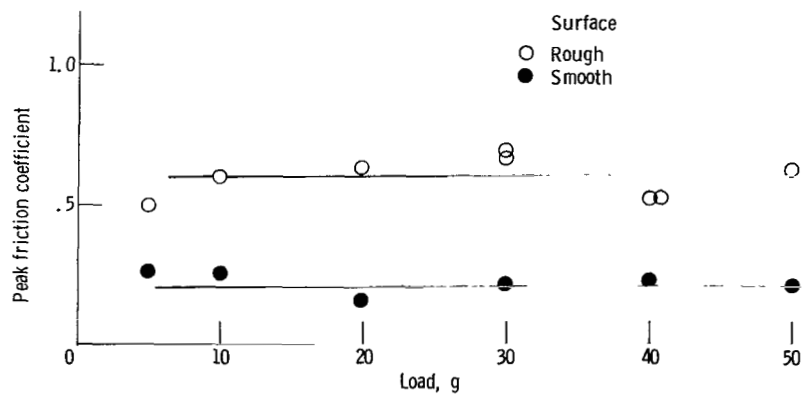


Figure 3. - Friction coefficient as function of load for titanium sliding on single-crystal silicon carbide (0001) surfaces in argon at atmospheric pressure. Sliding velocity, 3 mm/min; temperature, 25° C.

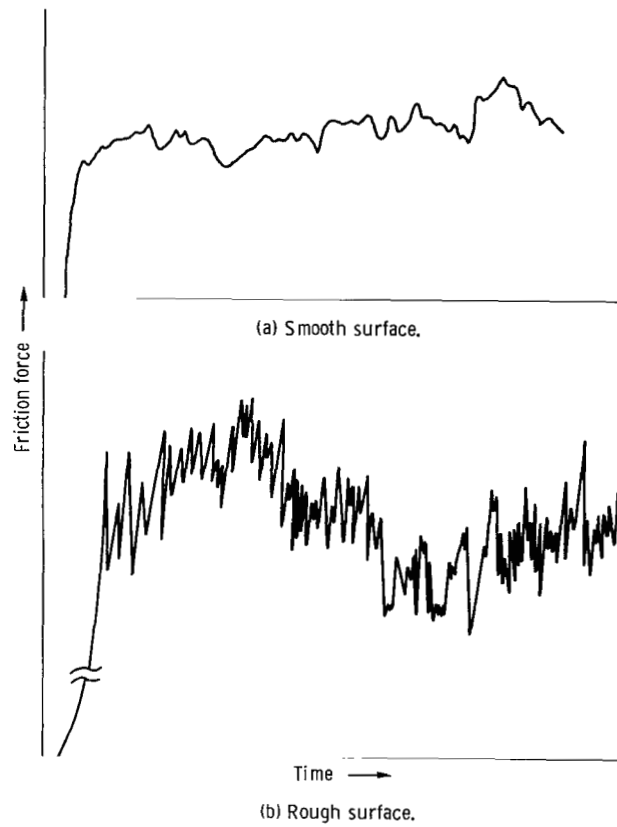
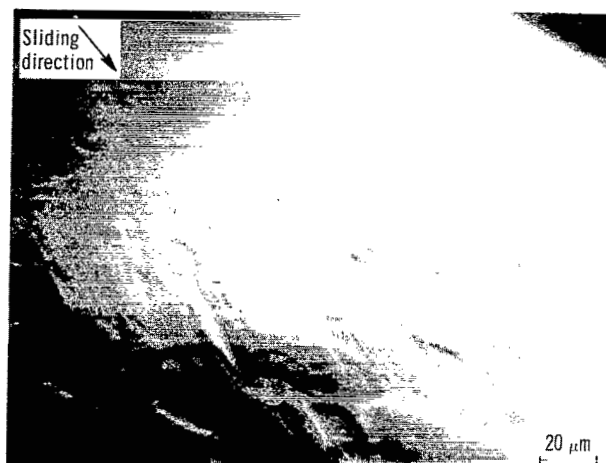


Figure 4. - Friction force traces for titanium sliding on single-crystal silicon carbide (0001) surfaces in argon. Sliding velocity, 3 mm/min; load, 20 grams; temperature, 25^o C; environment, argon; pressure, atmospheric.



(a) Beginning of wear scar.

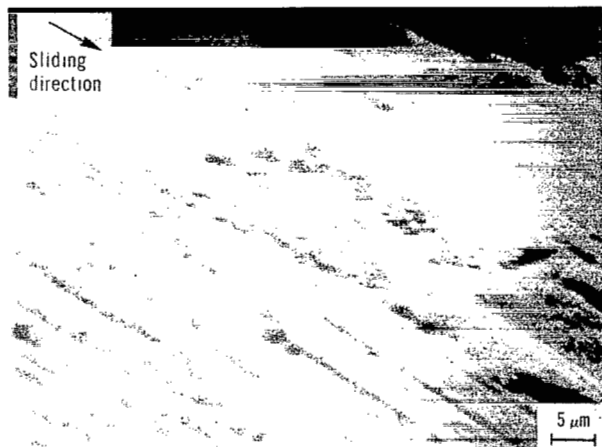


(b) End of wear scar.



(c) Silicon K_{α} X-ray map of titanium rider; 2×10^4 counts.

Figure 5. - Wear scar on titanium rider after sliding on smooth silicon carbide surface. Sliding velocity, 3 mm/min; load, 50 grams; temperature, 25° C; environment, argon; pressure, atmospheric.



(a) End of wear scar.



(b) Beginning of wear scar.

Figure 6. - Beginning and end of wear scar on titanium rider after sliding on smooth silicon carbide surface. Sliding velocity, 3 mm/min; load, 50 grams; temperature, 25° C; environment, argon; pressure, atmospheric.

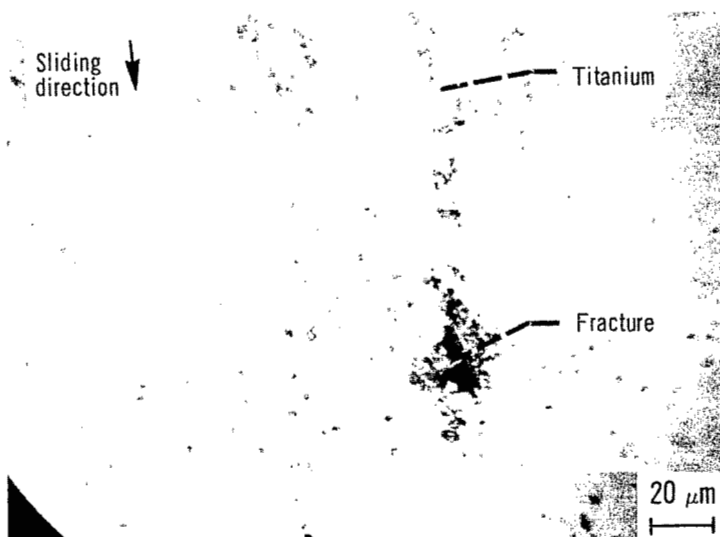
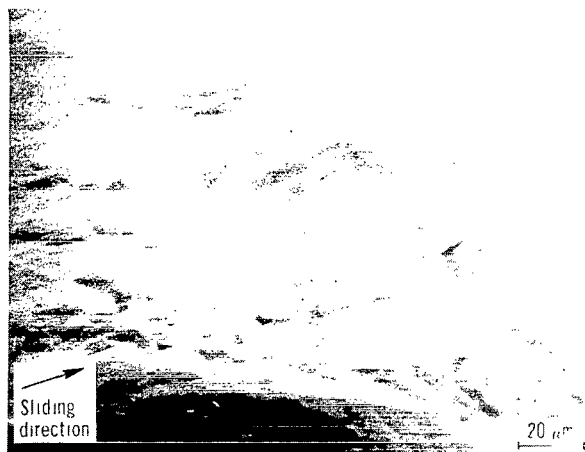
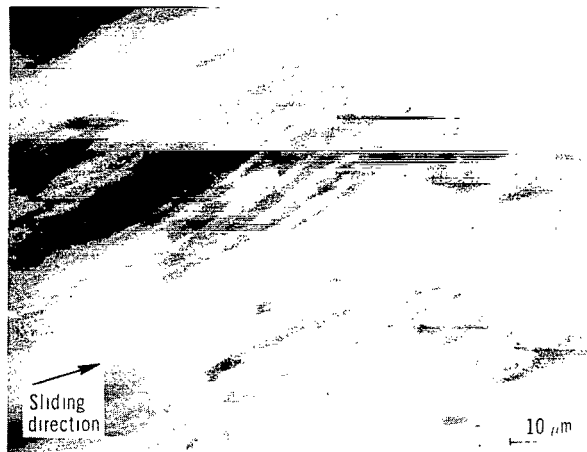


Figure 7. - Adhesion of titanium to smooth silicon carbide surface. Sliding velocity, 3 mm/min; load, 50 grams; temperature, 25° C; environment, argon; pressure, atmospheric. (Fracture occurs in silicon carbide.)

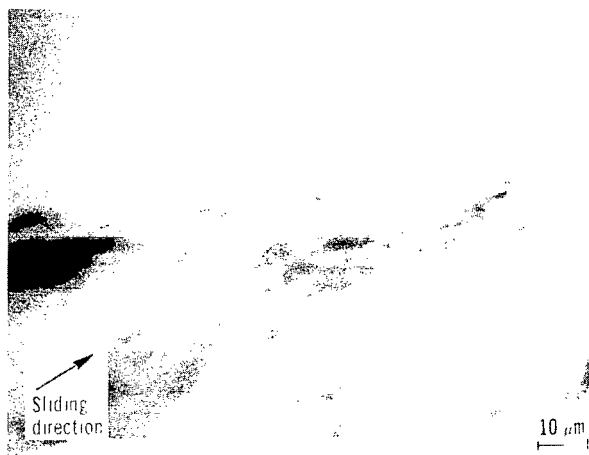


(a) Wear scar.

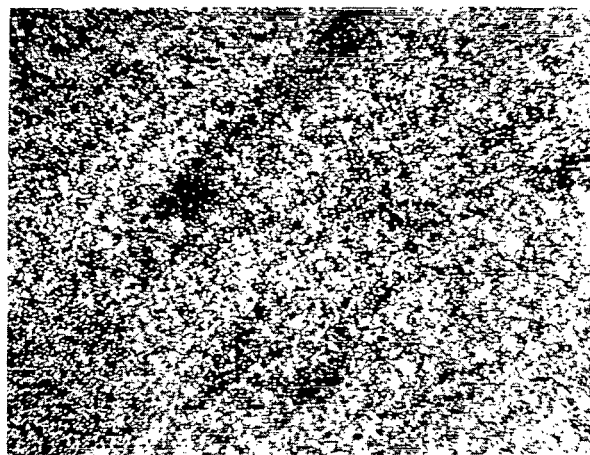


(b) Grooves in wear scar.

Figure 8. - Wear scar on titanium rider after sliding on rough silicon carbide surface. Sliding velocity, 3 mm/min; load, 30 grams; temperature, 25° C; environment, argon; pressure, atmospheric.

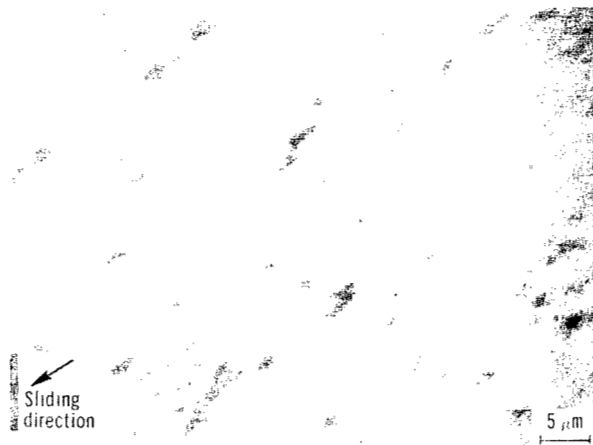


(a) Wear scar.



(b) Titanium K_α X-ray map of titanium rider; 4×10⁴ counts.

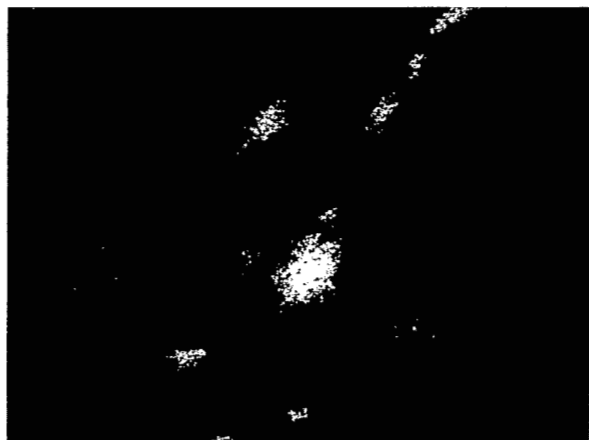
Figure 9. - Wear scar on titanium rider after sliding on rough silicon carbide surface. Sliding velocity, 3 mm/min; load, 30 grams; temperature, 25° C; environment, argon; pressure, atmospheric.



(a-1) Scanning electron micrograph.



(b-1) Scanning electron micrograph.



(a-2) Titanium K_α X-ray map of silicon carbide surface; 5×10^3 counts.

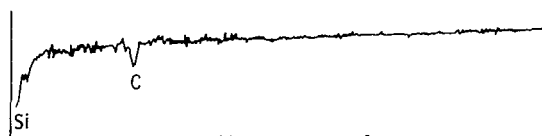
(a) Contact asperities.



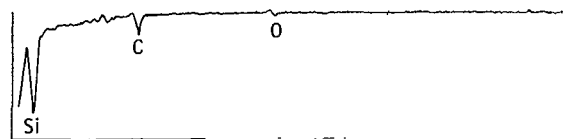
(b-2) Titanium K_α X-ray map of silicon carbide surface; 5×10^3 counts.

(b) Asperities capped with titanium.

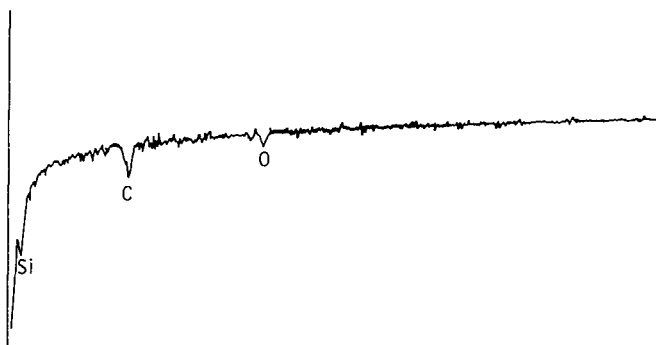
Figure 10. - Adhesion of titanium to asperities on rough silicon carbide surface. Sliding velocity, 3 mm/min; load, 30 grams; temperature, 25° C; environment, argon; pressure, atmospheric.



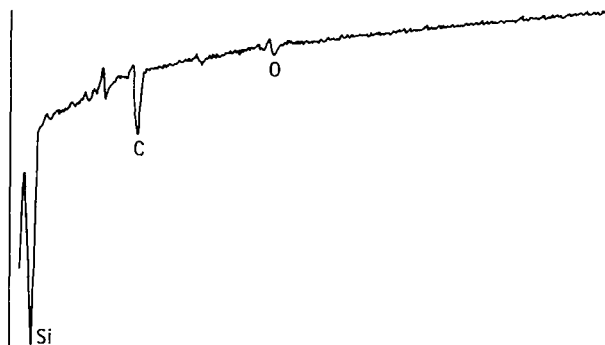
(a) Before sputter cleaning of smooth surface.



(b) After sputter cleaning of smooth surface.

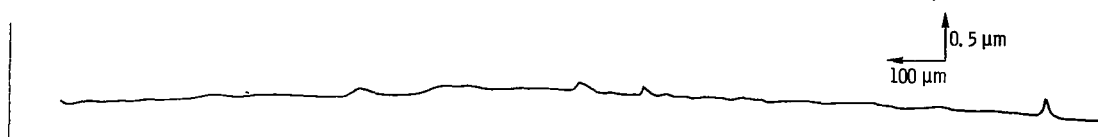


(c) Before sputter cleaning of rough surface.

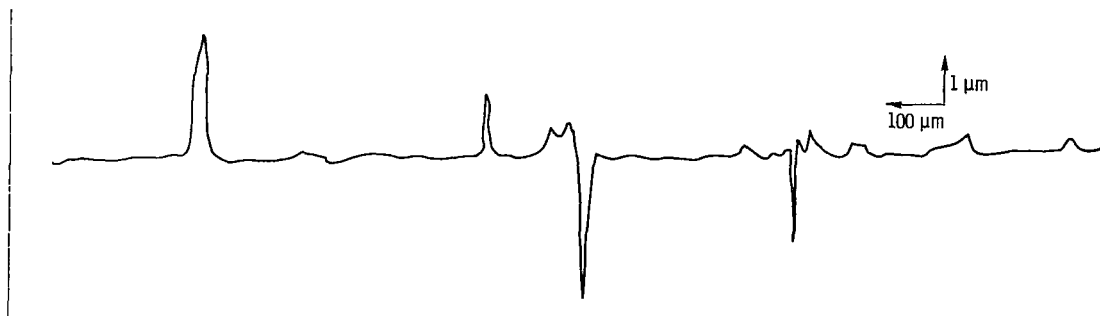


(d) After sputter cleaning of rough surface.

Figure 11. - Auger emission spectroscopy spectra for silicon carbide single-crystal surfaces.



(a) Smooth surface.



(b) Rough surface.

Figure 12. - Surface profile curves for silicon carbide after sputter cleaning.

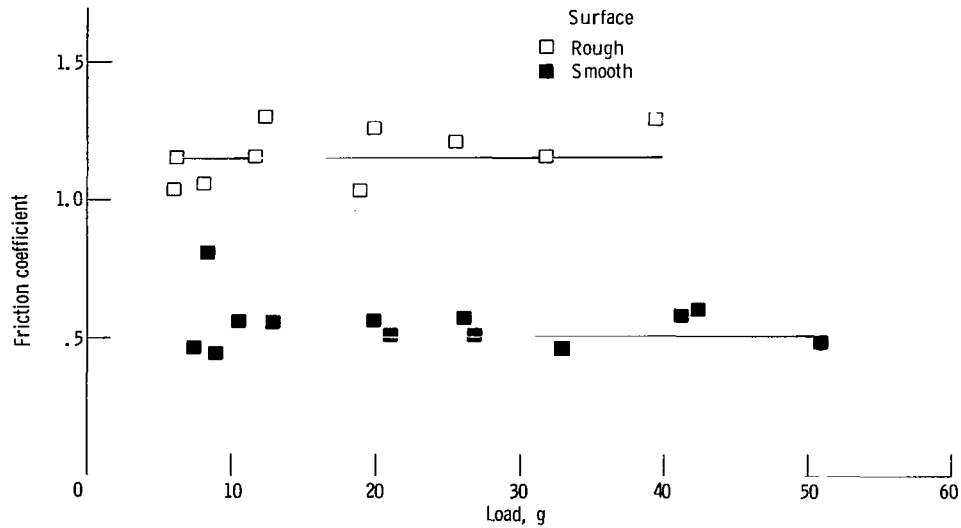


Figure 13. - Friction coefficient as function of load for titanium sliding on single-crystal silicon carbide (0001) surface in vacuum (10^{-8} N/m²). Sliding velocity, 0.7 mm/min; temperature, 25° C.

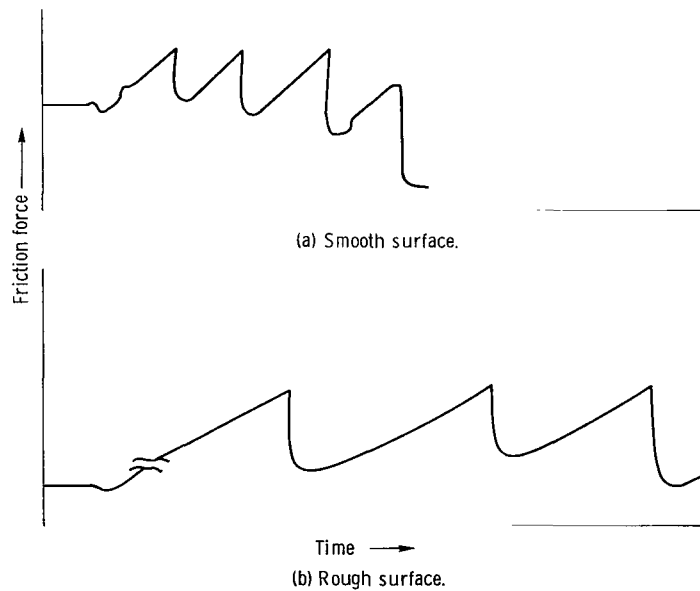
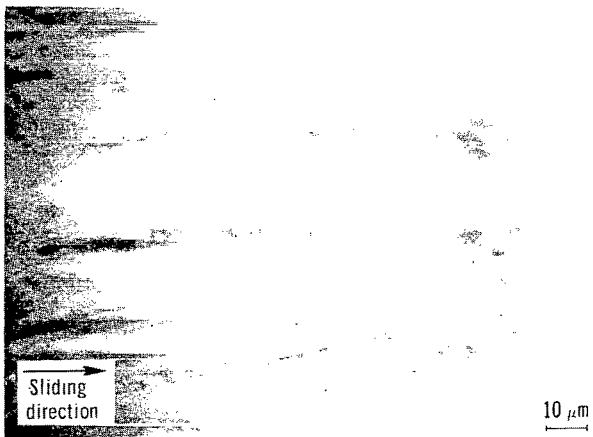


Figure 14. - Friction force traces for titanium sliding on single-crystal silicon carbide (0001) surfaces in vacuum. Sliding velocity, 0.7 mm/min; load 40 grams; temperature, 25° C; pressure, 10^{-8} N/m².



Figure 15. - Adhesion of titanium to smooth silicon carbide surface. Sliding velocity, 0.7 mm/min; load, 40 grams; temperature, 25° C, pressure, 10^{-8} N/m².

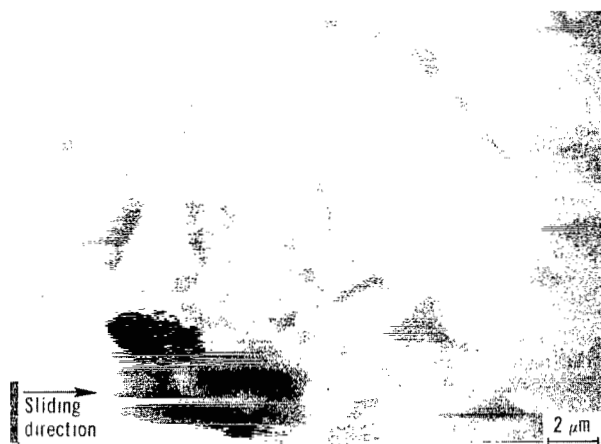


(a) Wear scar.



(b) Silicon K_α X-ray map of titanium rider; 5×10^3 counts.

Figure 16. - Wear scar on titanium rider after sliding on smooth silicon carbide surface. Sliding velocity, 0.7 mm/min; load, 40 grams; temperature, 25° C; pressure, 10^{-8} N/m².

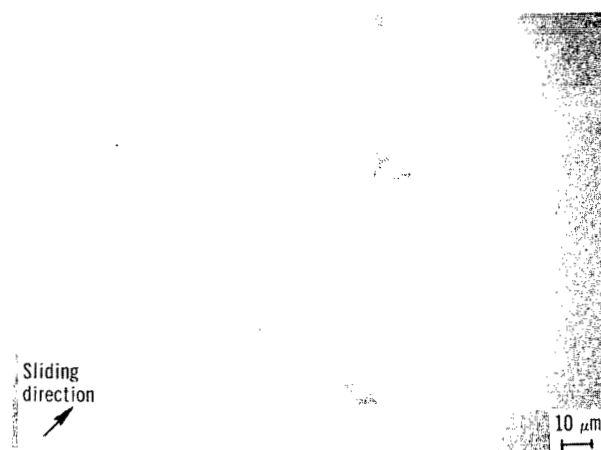


(a) Silicon carbide wear debris.

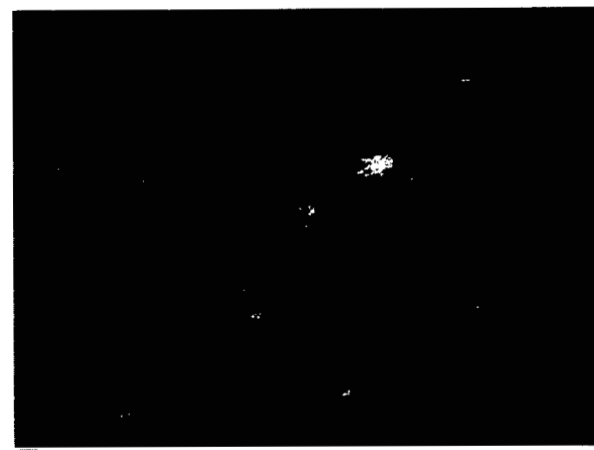


(b) Silicon K_{α} X-ray map; 5×10^3 counts.

Figure 17. - Silicon carbide wear debris on titanium rider. Sliding velocity, 0.7 mm/min; load, 40 grams; temperature, 25°C; pressure, 10^{-8} N/m².

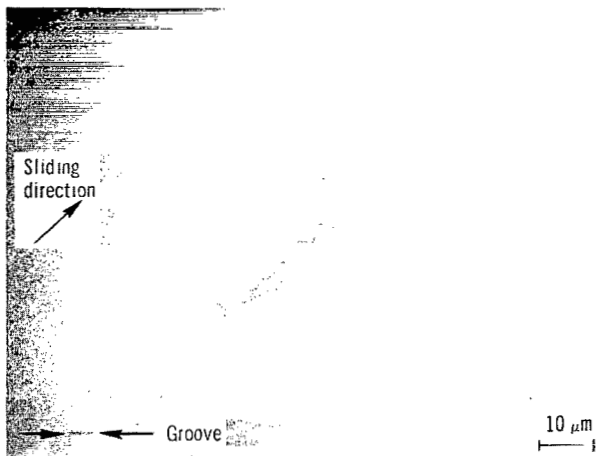


(a) Adhesion of titanium to silicon carbide.



(b) Titanium K_{α} X-ray map of silicon carbide, flat specimen; 5×10^3 counts.

Figure 18. - Thin film of titanium transferred to silicon carbide. Sliding velocity, 0.7 mm/min; load, 40 grams; temperature, 25°C; pressure, 10^{-8} N/m².



(a) Silicon carbide wear debris.



(b) Titanium K_{α} X-ray map of silicon carbide, flat specimen; 5×10^3 counts.

Figure 19. - Silicon carbide wear debris capped with titanium. Sliding velocity, 0.7 mm/min; load, 40 grams; temperature, 25° C; pressure, 10^{-8} N/m².

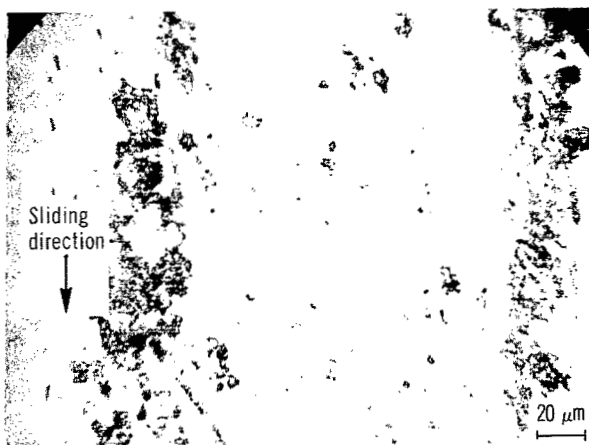


Figure 20. - Wear tracks on smooth silicon carbide surface. Sliding velocity, 0.7 mm/min; load, 40 grams; temperature, 25° C; pressure, 10^{-8} N/m².

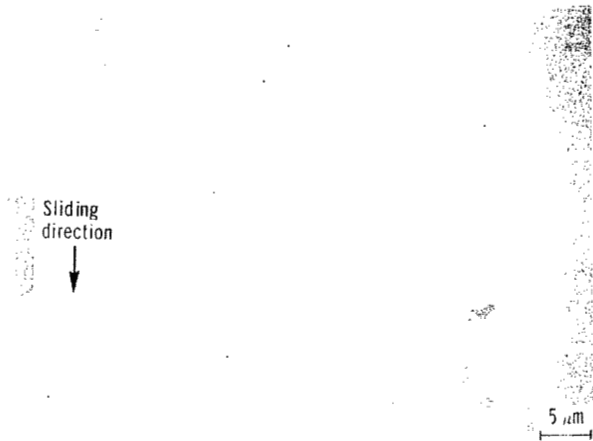
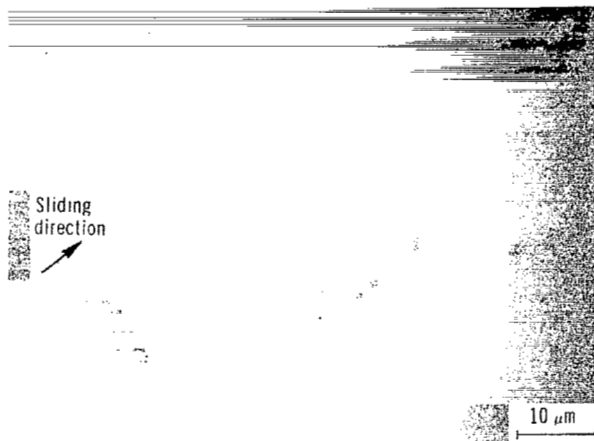
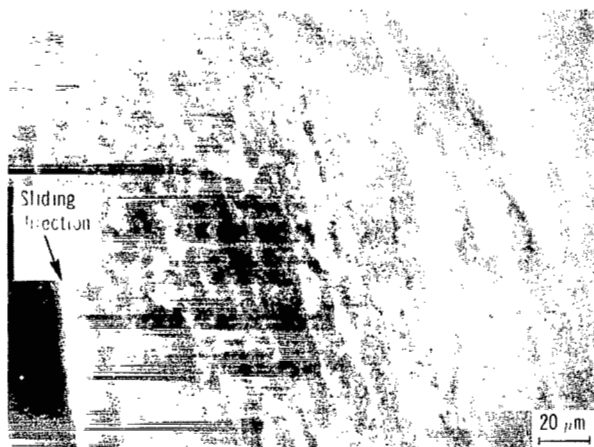


Figure 21. - Silicon carbide wear debris generated by fracture. Sliding velocity, 0.7 mm/min; load, 40 grams; temperature, 25°C; pressure, 10^{-8} N/m².



(a) Wear scar.



(b) Silicon K_{α} X-ray map of titanium rider; 5×10^3 counts.

Figure 22. - Wear scar on titanium rider after sliding on rough silicon carbide surface. Sliding velocity, 0.7 mm/min; load, 40 grams; temperature, 25°C; pressure, 10^{-8} N/m².

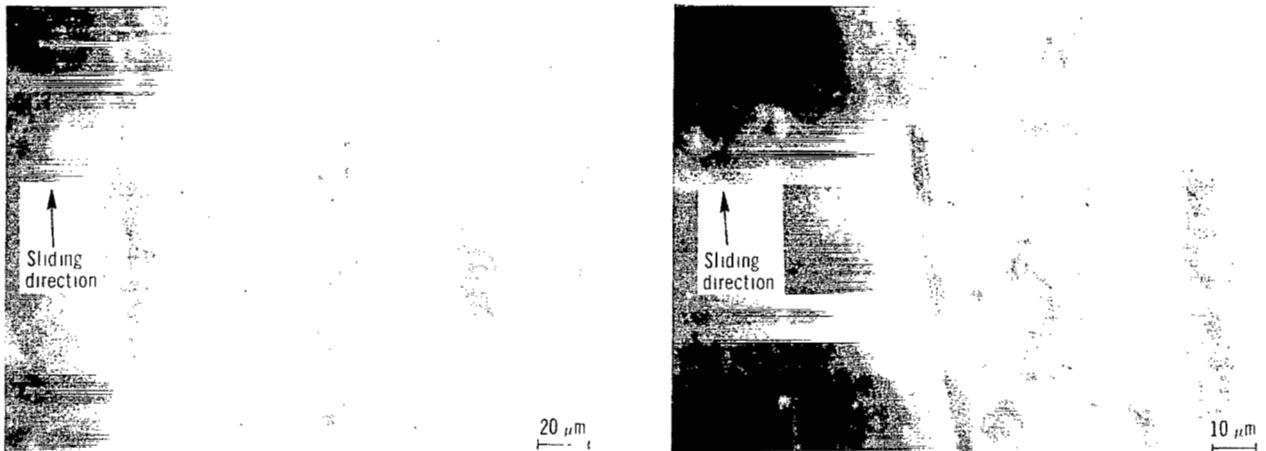


Figure 23. - Wear track on rough silicon carbide surface. Sliding velocity, 0.7 mm/min; load, 40 grams; temperature, 25° C; pressure, 10^{-8} N/m².

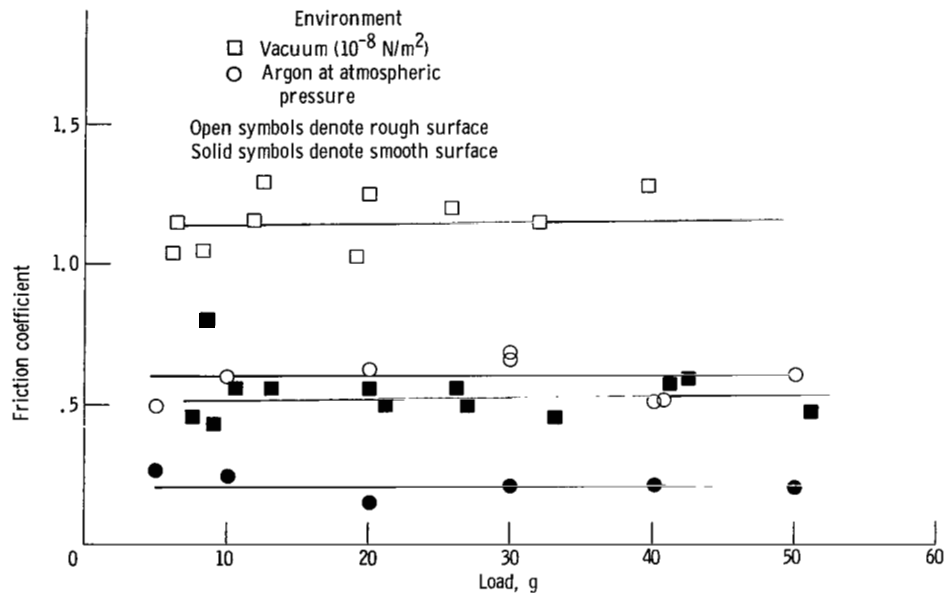


Figure 24. - Friction coefficient as function of load for titanium sliding on single-crystal silicon carbide (0001) surfaces both in argon at atmospheric pressure and in high vacuum. Sliding velocity, 3 mm/min in argon at atmospheric pressure and 0.7 mm/min in vacuum; temperature, 25° C.

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